APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

Review of World War 2 Methods of Manufacture and Storage for UK Sulphur Mustard (HT) Currently Held at Pine Bluff Arsenal, Arkansas, USA.

Richard Soilleux

Dstl/CR24879 V1 18th June 2007 Dstl Porton Down Salisbury Wiltshire SP4 0JQ © Crown copyright 2007 Dstl

Release Conditions					
	Da	0000	Can	ditio	no

This document was produced by Dstl for the US Army Chemical Materials Agency under US Army International Research Office Order/Contract reference N62558-07-P-0048

© International Research Office

	Name	Signature	Date
ESD Chief Scientist	Dr. Adrian Baker	Alfal	19/6/07
Project Manager	Eddy Doust	Cr /-	2 19/06/07.
Technical Reviewer	Dr. Robin Black	BIMO	18/06/07

Executive summary

The United States (US) is in the process of destroying stockpiles of mustard (HT) at its Pine Bluff Chemical Disposal facility (PBCDF), Arkansas. The agent is currently stored in "one ton" containers. It is important to fully understand the provenance and chemical composition of the agent being destroyed so that all risks can be fully appreciated and the safest, most efficient and environmentally acceptable disposal process can be used. The HT was thought to have come from different sources, either the Cornwall facility in Ontario, Canada or shipped from the UK in 1946 in No.6 Drums and 10 gallon British land mines.

This review has been carried out at the request of the US authorities in order to establish the provenance of the mustard and, if possible, determine whether the Canadian HT from the Cornwall facility, was manufactured in the UK. A further aim was to obtain the HT product specification and include any information on its composition or impurities that may be expected.

Many relevant documents have been found and, although there are clear gaps, most of the information requested has been retrieved. Detailed descriptions of the HT process have been identified together with significant amounts of information concerning impurities. In addition a large amount of information has been discovered on the construction of the Storage Drum, No.1, Mk 6 and the Mine Chemical No.1 Mk 1. It has not been possible to confirm details of the original shipment in 1946 of HT although a recent US report concerning this shipment has been found. Details have also been discovered of a separate, previously unknown, shipment of 350 tons of HT direct to Pine Bluff Arsenal in 1943.

Several reports were discovered of trials in Canada using HT of British origin so British HT was certainly shipped to Canada. Although no documents concerning the Canadian production of HT have been found in the UK archives, the US Army Chemical Materials Agency (CMA) has recently discovered Canadian documents that show that the Cornwall facility had two mustard plants, one producing Levinstein mustard (HS) and the other HT. It is therefore likely that the HT shipped to the US from Cornwall was of Canadian origin made to UK specifications. However, HT manufactured in the UK may also have been stored at the Canadian plant so a British origin, at least in part, for the "Cornwall" HT cannot be ruled out.

Table of contents

Execu	utive summary	1
1	Introduction	3
2	Scope and Limitations	3
3	Review	4
3.1	British Manufacturing Methods for HT used in World War 2 (WW2).	4
3.2	The thiodiglycol process for HT.	4
3.3	UK Factories that made HT.	5
3.4	Precursor Chemicals.	6
3.5	Changes in the process.	6
3.6	Impurities.	6
3.7	Thickened HT.	7
3.8	Storage and transport containers	8
3.9	Shipping records	10
3.10	Canadian information	11
4	Discussion	14
5	Conclusions	16
6	List of references	17
7	Acknowledgments	19
Initia	al distribution	21
Repo	rt Documentation Form	23

1 Introduction

The United States (US) is in the process of destroying stockpiles of the chemical warfare (CW) agent HT, a mixture of sulphur mustard (H) and an oxygenated homologue (T), at its Pine Bluff Chemical Disposal facility (PBCDF), Arkansas. The agent is currently stored in "one-ton" containers. It is important to fully understand the provenance and chemical composition of the agent being destroyed so that all risks can be fully appreciated and the safest, most efficient and environmentally acceptable disposal process can be used. The HT is thought to have come from a combination of three sources:

- a. The Cornwall facility in Ontario, Canada.
- b. Shipped from the UK in 1946 in Mark 6 Drums.
- c. Shipped from the UK in 1946 in 10 gallon British land mines.

1.1 Aims

The aim of this review is to establish the provenance of the HT and, if possible, determine whether the Canadian HT was manufactured in the UK. The review also aims to obtain the product specification (including what thickening agents were used), or specifications (if it changed), and include any results from the historical analysis of HT or its reaction products or impurities. If it is shown that the Cornwall HT was produced in Canada, an additional aim is to determine if the Canadians were using the same specifications and processes for producing HT as the UK.

1.2 Scope and Limitations

This report is based on information discovered in the British archives during the search period. A significant quantity of information has been identified however, there are still some gaps. In particular, little information was discovered on the provenance of the Canadian HT.

2 Review

2.1 British Manufacturing Methods for HT used in World War 2 (WW2).

Several manufacturing methods for producing mustard were in use during WW2 in order to maximize production with the plant and starting materials available. For completeness, these methods are summarized below before the HT process is discussed in more detail.

- a. The sulphur monochloride process [1]. Ethylene and sulphur monochloride react to give H and sulphur. This was the basis of the American Levinstein and the South African DESA (for Defence Establishment South Africa) processes. The sulphur monochloride process was used by UK during WWI and developed between the wars. It was eventually decided that the process was unsatisfactory and it became obsolete in UK.
- b. The pyro or ethylene-sulphur dichloride process [1]. This was much more rapid than the monochloride process. No sulphur was formed thus avoiding the main complication of the monochloride reaction. The process was capable of producing good quality H but difficulties were encountered because of impurities in the sulphur dichloride.
- c. The thiodiglycol process for HT [1]. HT was prepared by interaction of thiodiglycol and hydrochloric acid. The mixture of H and T had the advantages of being stable in storage, of lower freezing point than H, and more vesicant and persistent than the pure agent. Largely for these reasons, HT became the favoured British vesicant agent in WW2 and was produced in large quantities.

2.2 The thiodiglycol process for HT.

2.2.1 Introduction.

The following section summarises the most relevant information available on the production process for HT that was in use in UK during WW2. (HT was also known as Runcol after Runcorn in Cheshire where much of it was made at the Rocksavage factory.) The process is complex, however, and much technical detail is available in the old reports, some of which may be helpful to fully understand the chemistry. To avoid the possibility that important information concerning potential contaminants has been missed in the summarizing process, the most relevant reports have been copied and will be made available with this report for further study. Also provided with this report is a DVD, which contains a copy of a training film produced for the factory workers at the Ministry of Supply (MOS) factory at Valley. There is not much technical detail in the film but it gives a good idea of the plant, methods and conditions of working at the time and it is hoped that it will help to put the written material into context.

2.2.2 Summary of the HT process.

HT is a mixture of H and T.724, which is bis(2-chloroethylthioethyl) ether, prepared by the interaction of thiodiglycol (TDG, also known as syrup which also contained some water) and hydrochloric acid at about 110°C [1]. Variation in the conditions of reaction, of which temperature is the most important, gives rise to some variation of the composition of the product. The normal product was a nominal H60/T40 mixture although the "T" part contained appreciable quantities of the homologues of T.724 and related compounds.

Gaseous hydrogen chloride was made from aqueous hydrochloric acid and concentrated sulphuric acid. The spent acid was concentrated to recover the sulphuric acid. Hydrogen chloride, under slight pressure, was passed into the preheated TDG mixed with a proportion of aqueous hydrochloric acid. On completion of the reaction, the mixture was transferred to a settler. The product was separated from the aqueous layer and it was dried by warming under reduced pressure.

The pressure stability, in storage containers and munitions, of HT was better than that of any other production H obtained on a large scale, and this was its main advantage over other production methods. Also, since T is about 3 times more vesicant than H, the vesicant power of the mixture was greater than that of pure H and significantly greater than other mixtures of H mixtures in use at the time, which contained inert diluents in order to reduce the freezing point for winter use. The low temperature behaviour of HT is demonstrated by a test of production 60/40 HT [2] which showed that although the melting point was 1.8°C, it was "quite fluid" at -10°C and "a soft mush" at -15°C.

2.3 UK Factories that manufactured HT.

The factories that have been confirmed to have produced HT are as follows. Sutton Oak, at St Helens in Lancashire [3] (although this was a Research and Development Establishment, the HT pilot plants, including the 50-ton/week HT plant, produced material that was added to the stockpile [4]); Randle, Springfields, Lancashire [5]; Valley, near Mold in North Wales; and Rocksavage, Runcorn, Cheshire. All these plants were known to have produced other types of mustard (mainly HMD, HM is the designator for mustard made by the sulphur dichloride process and HMD refers to an improved method where the sulphur dichloride was distilled in glass) but between them they produced all the British production of HT as well. Each of the main plants produced up to 144 tons/week with a total wartime production for the UK of 42,234 tons of HMD and HT [5].

There does not appear to be any records remaining that show details of what was produced at each factory, let alone individual production batches, or where they were sent. It is known, however, that storage capacity at some of the factories was limited and the mustard was moved into secure storage as soon as possible because of the heavy German bombing of British industrialized areas. The main bulk storage for mustard was at Valley in specially constructed underground tunnels and at Randle in underground "mustard pots" and tanks in bunkers. Much of the production was also dispersed to four "Forward Filling Depots" (FFDs), with large 200-ton underground concrete mustard pots, for use by the Royal Air Force (RAF). There were also two US depots with similar

mustard pots that were supplied by the UK (see paragraph 3.4). Although the different types of mustard were kept separate it is likely that different batches of each type were bulked up in the storage pots with no particular effort to avoid mixing materials from different factories.

2.4 Precursor Chemicals.

Precursor chemicals came from a number of sources but mainly ICI factories [5]. TDG was made at Rocksavage and Randle as well as another Ministry of Supply (MOS) Factory, Wade at Middlewich in Cheshire, and there may have been others. Some TDG was obtained from the US. The TDG for HT production at Sutton Oak was "purchased" [3]. HCl gas was made on site from concentrated HCl purchased from the chemical industry and probably came from a number of sources. Precursor chemicals from each of these sources would have had slightly different compositions due to unavoidable variations in process and raw materials. No records have been found (and they probably do not exist) which describe the various sources of precursor chemicals or variations in impurities.

2.5 Changes in the process.

The process was piloted at Sutton Oak before full-scale production started at Randle. It does not appear that the basic method changed during the whole of the production period although HT was made at more than one factory and there are bound to have been local variations in equipment and procedure. There is mention in reference [5] that "on the basis of operating experience (8 months, starting 15th February 1940), various minor modifications in the process and to a number of plant items have been made. These modifications have been introduced into the design of further Runcol plants now being constructed at MOS factory Valley".

2.6 Impurities.

Known and potential impurities from the production of HT are discussed in the following sections and summarized in Table 1. Early workers experienced problems arising from the purity of TDG made by the chlorohydrin process but these were later overcome when it was prepared from ethylene oxide [4]. For example, some batches of TDG were heavily discoloured, and development work had to be undertaken to investigate the cause and changes made to correct the problem [6]. It was concluded that the discoloured syrup (TDG) gave similarly discoloured Runcol (HT) which made difficult the separation of Runcol and toxic acid. Poor separation at this stage then lead to the introduction of sulphonium chlorides into the product resulting in pressure development on storage. It was recommended that the sodium hydrosulphide content of the sulphide liquor should be maintained at 1-1.5%. It is likely, therefore, that some of this sodium hydrosulphide was carried over into the finished product.

There is not much more information available on production impurities but one report has been found [7] that describes experiments and measurements that were undertaken in an attempt to identify the principal impurities in plant HT. These experiments centred on analysis of the fraction of the HT mix containing T. A rough estimate of the amount of

Q, or sesqui mustard (1,2-bisC2-chloroethy(thio)ethane) in the T fraction was made by two methods (a) by direct fractionation at low pressure and (b) by oxidation and isolation of Q sulphone from the products. The latter method gave a value of 10% Q in T (4% in HT) and it was thought that this was probably accurate to within ±2%. The results of the former method, 3.5% Q actually isolated in a state of purity from plant T are not inconsistent with this figure. The distillation residue from plant T was also examined and found to consist mainly of an ether insoluble chlorine-free solid, m.p. 75-80°C and of molecular weight of about 630. It was not possible to purify this product and it was not further identified. It was shown, however, that it was not present in quantity in plant T but was formed during distillation, the amount depending on the time of heating and the temperature used.

Reference [4] describes the operation of the 50-ton/week plant at Sutton Oak and reports that a typical bulk product containing 58.1% H and 41.2% T had about 0.7% volatile matter.

Reference [5] contains the detailed specification for the Runcol but this is based on permissible ranges for the concentrations of the principle components and physical measurements such as specific gravity and did not require identification and analysis of impurities.

The construction materials for Sutton Oak and Randle HT plants are recorded in references [3] and [5] and the contaminants likely to have arisen from these and carried over into the final product are discussed in section 3.3 and listed in Table 1. However, details of the instrumentation for the Sutton Oak plant were not recorded and those for the Randle plant are in a separate document which has not been found. It has therefore been assumed that the instruments would have been made of similar materials to the rest of the plant and would not therefore introduce additional contaminants, except that thermometers and manometers containing mercury would have been used. These may have occasionally broken to introduce mercury into some batches in an unpredictable way so this potential contaminant has been included in Table 1.

2.7 Thickened HT.

A number of different thickening were added to British weaponised mustard, including HT, in order to improve its dissemination characteristics, especially when air-burst. No indication has been found that the HT supplied to the US during WWII contained thickening agents. (Indirect evidence for the lack of thickening in US stocks of HT comes from recent experience in demilitarising HT. Work in the two countries has shown that whereas US "heel material" which consists largely of complex mixtures of sulphonium ions breaks up readily in hot water, British "heel material", which often contained inert thickening agents, does not.) Nevertheless, a review to determine if HT shipped to the US had been thickened, and what methods may have been used, was requested by the US. This was to cover the unlikely event that thickened HT may be encountered in the US stockpile because, if thickening had occurred, British methods and materials would almost certainly have been used. The British thickeners for mustard have been reviewed and summarized below.

The most common materials used to thicken mustard were chlorinated rubber, plexiglass (polymethyl methacrylate) vinyl acetate or Gelva (a low molecular weight polyvinyl acetate) [8]. There were problems in maintaining HT thickened with chlorinated rubber or vinyl acetate of consistent viscosity on prolonged storage in steel containers, even when varnished, largely due to the presence of small amounts of ferrous chloride [9] which tended to promote an increase in viscosity leading ultimately to gelling. It was further shown that the iron content of HT varied widely (factor of 10) depending on batch and the factory in which it was made [14]. HT from Sutton Oak seems to have the lowest iron content at about 5 parts per 100,000, by weight, whereas samples of Randle HT had 53 parts per 100,000. It was recognized that the iron content would tend to increase because of the action of traces of hydrochloric acid on the steel storage containers despite the protecting effect of the varnish which could never be 100%. It was also noted that "Randle HT, judged merely by colour, has shown marked fluctuations in quality, with, on the whole, a tendency to improvement" [10]. (This observation was made in 1941 so it is to be hoped that the shipments made to the US in 1943 and 1946 were of better quality.) In an attempt to minimize the production of ferrous chloride, various stabilizers were tried including magnesium oxide, hexamine, and dimethyl aniline [9].

It is considered unlikely that thickened HT will be found in the US stockpile. However, should some be present the thickeners and stabilisers listed in Table 1 may have been used.

2.8 Storage and transport containers

2.8.1 General

Brief descriptions of the containers used for storage and transport of the HT to the US follow with particular reference to materials of construction that could have caused contamination of the contents. Although the Drum No.6 is documented, no references have been found to a 10-gallon land mine, as described in the US contract documents, and the description of this mine is thought to be in error. There are, however, many references to the Chemical Mine No.1 (Figure 3.2) which has a capacity of 3.7-gallons (UK gallons are 4.5 litres) which is of very similar construction to the Drum No.6 (Figure 3.1) and it is thought likely that these were the ones used and that the 10-gallon mine did not exist. This conclusion is supported by comparing the photographs for the Drum No.6, supplied by the US authorities, with UK photographs of the Chemical Mine No.1.

It is worth noting, however, that a number of storage and transport containers for mustard were in existence at the time ranging from 1-pint and 1-gallon (UK) earthenware jugs and 1-pint steel containers up to 90-gallon (1054lb or 479kg) steel drums [11]. Except for the earthenware jugs, which were not likely to have been used for transport to the US because of their small size, any of these containers could have been used but as they were of very similar materials of construction (leading to the same range of contaminants in the mustard) the following has been limited to descriptions of the Drum No.6 and the Chemical Mine, No.1.

There do not appear to be any records remaining of where the various munitions and containers were filled, not even whether they were filled at the factory direct from current production or whether they were filled from bulked up stored agent. It seems likely, however, that once agent had gone into bulk storage, especially in RAF or US depots, it would have remained there and new requisitions for agent filled from current production. It would explain the difference in physicochemical properties seen in different ton containers in the remaining US stocks, if they had been filled from different batches of HT, possibly from different factories.

2.8.2 Drum Storage No.6 MkI

The Drum, Storage, No.6, Mk. I, was made up of a cylindrical body which tapered to a cylindrical neck [12] [13]). The diameter of the cylindrical body was 11.12 in., and that of the neck 3.5 in. The height of the cylindrical body was 11 in., that of the neck 3.5 in., and the overall height 18.35 in. The capacity of the drum was 4.6 gallons (UK) but the normal amount of charging was 3.7 gallons (16.65 litres). The neck was closed by a screwed stopper, fitted with a Nordoil (no information has been found on the composition of this material) washer. The threads of the stopper were coated with C.D. cement No.3 (red and white lead / boiled linseed oil [14]), and the stopper was secured in position by a length of copper wire passed through holes in the stopper and under a staple on the body of the drum.

The drum was constructed of terne-plate (mild steel sheet coated with an alloy of tin and lead [15]), and its internal surfaces coated with Bakelite varnish. (Bakelite is a trade name for a phenol-formaldehyde resin which can be used as a varnish. It is a brittle material best described as an irreversible thermosetting resin). It is not clear whether the seams of the drum were welded or soldered but, if it was constructed from terne-plate as described in the reference rather than being plated afterwards, the latter method is most likely. A photograph of the drum provided by the US authorities is shown below in Figure 3.1.



Figure 3.1 – Drum, Storage, Number 6 Mk 1

2.8.3 Mine Chemical No.1, Mk.I

The Mine, Chemical. No.1. Mk.I. B.V., consisted of a lead coated steel drum of commercial pattern, varnished internally (B.V. in the designation stands for Bakelite

Varnish) [16] [17]. Although the description in the reference is for steel coated with lead this does seem likely since lead does not alloy with steel and it is probable that a tin lead alloy was used as it was for the storage drums. The Chemical Mine (Figure 3.2) was of very similar appearance to the Drum Storage No.6 (Figure 3.1) described above with the same dimensions and a capacity of 50-pounds (3.75 UK gallons).

The main difference between the mine and storage drum was the provision of a burster well, 2-inches in diameter and 13 inches long, in the mine. The explosive bursters were intended to be inserted just before use so it is most unlikely that bursters would have been fitted to the mines if they had been used as shipping containers. The construction and composition of the bursters has therefore been ignored except to say that the cast iron stopper was sealed with a leather washer to close the neck of the drum.

Likely contaminants of the HT from the storage drum and chemical mine are iron, tin and lead with traces or organic compounds from the linseed oil and Bakelite varnish. These are listed in Table 1.



Figure 3.2 - Mine, Chemical. No. 1. Mk.I.

2.9 Shipping records

A limited amount of information has been found concerning the shipment of HT from UK to the US. A relatively recent (1987) US document reporting the shipment from Cardiff on the SS George Walton to Charleston SC in 1946 has been found [18] but it has not been possible to confirm this from original UK sources.

However, of particular interest are the War Office and Ministry of Supply Contract Record Books which, among other things, record all quantities of HT supplied to various customers during WWII. One of these (contract No. 294/7/4244 placed on the 17th

September 1943) was for 350 tons of HT 60/40 that was to be delivered direct to Pine Bluff Arsenal, Arkansas [19]. The order was placed on "Ministry of Supply Factories" which were not individually identified so it is possible that the delivery was made up from several sources. Very little more information is given except that that the delivery was to be in drums (type not specified) which were costed separately at £4,000 (the HT cost £46,000).

A number of other orders were made by the US Army for HT (totalling 5,000 tons) and recorded in the Contract Record Books [23] but all deliveries (all from the Rocksavage, Runcorn factory) appear to be to US bases in UK (Melchbourne Park, Bedfordshire and US Army Depot Weatherby, Shropshire). Some of the deliveries to Melchbourne Park were to be in re-usable 5 ton containers whereas the delivery to Weatherby was in 55 gallon drums provided by the US authorities. Documentation has also been found which throws doubt on whether all of these orders were filled (4,500 tons were cancelled but more orders were expected). However, this falls outside the scope of the review and no attempt has been made to confirm this.

It may be that the HT shipped to the US on board the SS George Walton was that supplied to the UK based US forces in 1943. If the US forces then shipped this material to the US it may explain why no record has appeared within the UK archives.

It is worth noting that in British WWII documents, thickened HT is designated as HTV where the "V" stands for viscous. Since this designation was not used in the Supply Contract Record Books for any of the shipments sent to the US where it was always referred to as HT, this is good evidence that the thickened version was never supplied.

2.10 Canadian information

It has not been possible to confirm from UK records that HT was manufactured in Canada at the Cornwall plant or anywhere else. References have been found [8] and [20] to HT being used in trials in Canada but, in one case at least, the HT was clearly stated to be of British origin "The sample in question was shipped by boat from UK in June 1941" [20].

On the other hand, reports have been found referring to Canadian work on the purification of American Levinstein mustard [21] and even the construction (in 1944) of a pilot plant for the purification of Levinstein mustard [21].

Information (attached) recently discovered by the US Army Chemical Materials Agency (CMA) indicates that the Canadian facility operated by Stormont Chemicals in Cornwall, Canada did, for a brief period (13 months, probably during 1943 - 44) manufacture HT [22], [23] and [24]. Reference [22] suggested, in March 1943, that Candian experts should be sent to UK to "familiarise themselves with the British manufacturing process" so it seems likely that the same process was used in both countries. There is anecdotal evidence [24] that shipments of mustard, type not specified, were made to the US "some of the products of the Stormont Chemical Company were sent to the US as well. Many tonnes of gas were sent to the Red Stone Arsenal in Huntsville, Alabama".

It is therefore possible that some of the HT stored at Cornwall, Canada had been manufactured in UK, while the Stormont Chemical Company using the UK process manufactured other batches of HT. It is not possible to say from the information available whether the shipments to the US were of Canadian or British HT or a mixture of the two.

Process leading to contamination	Source of contamination	Type of contamination	Comments
Precursor chemicals		HCl	Residual starting material
		Water	Left over from hydrochloric acid
		Sulphonium chlorides	Reaction products
		Sodium hydrosulphide	Residual starting material
		Q, sesqui mustard	By-product
		Unidentified solids	Polymer by-products
		Unidentified volatiles	Solvents
Plant construction materials	Cast Iron (C.I.) Reaction/storage vessels	Ferrous chloride	From reaction with HCl
	Mild Steel (M.S.) pipework	Ferrous chloride	From reaction with HCl
	Asbestos gaskets	Asbestos fibres *	
	Regulus metal pumps	Antimony and lead chlorides	From reaction with HCl
	Lead pipework	Lead chloride *	From reaction with HCl
	Ebonite	Sulphur *	Highly vulcanized rubber
	Rubber tubing	Rubber in solution	
	Tiles/ earthenware	Lead chloride *	Leached from lead salt glazed tiles
	Thermometers and manometers	Mercury chloride *	From reaction with HCl
Thickening	Thickeners	Chlorinated rubber *	Very unlikely in US HT
		Vinyl acetate *	Very unlikely in US HT
		Polyvinyl acetate (low MWT) *	Very unlikely in US HT
		Polymethyl methacrylate *	Very unlikely in US HT
	Stabilisers	Magnesium oxide *	Very unlikely in US HT
		Hexamine *	Very unlikely in US HT
		Dimethyl aniline*	Very unlikely in US HT
Storage/ containers	Tern plate	Lead and tin chlorides*	From reaction with HCl
	Solder	Lead and tin chlorides*	From reaction with HCl
	Cement CD No.3	Lead chloride*	From reaction with HCl

Table 1 - Known and potential (*) contaminants of British WWII HT.

3 Discussion

The amount of information on which this report is based has been limited to what has been found during searches in the British National Archives, Dstl archives and other sources. The historical information is in the form of old documents, mostly in hard copy and even those that have been scanned are filed in a somewhat haphazard manner. The search process therefore depends largely on the individuals concerned developing a sense of the types of files to be searched and there is some degree of luck involved. Nevertheless, many relevant documents have been found and, although there are clear gaps, most of the information requested has been retrieved.

Detailed descriptions of the HT process have been found and significant amounts of information concerning impurities. Although the information on impurities is not as detailed as one would wish from a modern perspective, what has been retrieved is probably as complete as it can ever be. Indeed, it is highly likely that more accurate and comprehensive analyses for impurities were never carried out given the wartime pressures to produce agent in sufficient quantity, with quality only being of interest if it affected performance. This last point is illustrated by the efforts that were expended to solve problems due to the pressure build up caused by gas producing corrosion reactions within steel containers and the problems that some impurities caused with thickened mixtures. For convenience, all information on known and likely contaminants has been collected and summarized in tabular form (Table 1). Copies of the original documents have also been made available to give as complete a picture as possible and to allow the engineers involved in the HT disposal operation to study the details of the production process.

Detailed information has been discovered on the construction of the Storage Drum, No. 1, Mark 6 and the Mine Chemical No 1 Mk I. They were both based on the same 3.75-gallon (UK) sheet steel container protected on the inside with a tin/lead alloy coating and Bakelite varnish. Nothing has been found concerning a British 10-gallon mine which does not appear to have existed so the reference to this in the American documentation is probably an error. This is supported by the fact that a photograph of the land mine referenced in the US report is identical in appearance to the Storage Drum No.1, Mark 6 and the Chemical mine, No1, Mk1.

It has not been possible to confirm details of the original shipment in 1946 of HT although the 1987 US report concerning the movement has been found. It appears in this case that the US has retained some records that have either been destroyed in the UK or not found during the 4-week search period. Details have been found, however, of a separate, previously unknown, shipment of 350-tons of HT direct to Pine Bluff Arsenal in 1943.

Although several reports were discovered of trials in Canada using HT of British origin, nothing at all has been found concerning the production of mustard by any process greater than laboratory scale. Reports have also been found which describe Canadian efforts to purify Levinstein mustard of US origin which culminated in the construction of a pilot plant for solvent extraction of mustard from the crude reaction product. It is

known that mustard was produced in Canada at the Cornwall plant near Ottawa, but it has not been possible to find any information in the UK archives on the plant, the process used and the type of mustard produced. However, the US Army Chemical Materials Agency (CMA) has recently obtained documents that show that the Canadian facility operated by Stormont Chemicals in Cornwall, Canada manufactured both Levinstein Mustard and HT. The documentation suggests that the manufacturing process developed in UK was also used in Canada. There is anecdotal evidence that shipments of mustard, type not specified, were made to the US, specifically to Red Stone Arsenal in Huntsville, Alabama from Canada.

It therefore seems likely that HT manufactured by the British process by Stormont Chemical Company in Cornwall, Canada was shipped to the US. However, it is possible that some HT of British origin was stored at Cornwall and it is not possible to say, from the information available, whether the shipments to the US were of Canadian or British HT or a mixture of the two.

4 Conclusions

Many documents relevant to WW2 production of HT have been found and, although there are clear gaps, most of the information requested by the US authorities has been retrieved.

Detailed descriptions of the HT production process have been found together with significant amounts of information concerning impurities.

Detailed information has been discovered on the construction of the Storage Drum, No. 1, Mark 6 and the Mine Chemical No 1 Mk I.

It has not been possible to confirm details of the original shipment in 1946 of HT although the 1987 US report concerning the movement has been found. Details have been found, however, of a separate previously unknown shipment of 350-tons of HT direct to Pine Bluff Arsenal in 1943.

Several reports were discovered of trials in Canada using HT of British origin but nothing has been found in the UK archives concerning the production of mustard by any process at greater than laboratory scale. However, Canadian documents obtained recently by the US indicate that some production did occur. It is therefore possible that some of the HT shipped to the US from Cornwall in Canada was of British origin while some was of Canadian manufacture to British specifications.

5 List of references

- 1. A survey of Processes for the Manufacture of H. SO/R/623, 31st December 1942.
- Extract from Sutton Oak Report SO/R/523, on the consistency of HT 60/40 at low temperatures.
- 3. Sutton Oak Report S.O. 481, Production of HT, Description of Process and Plant, 16th October 1940.
- 4. The Classical Chemical Agent Handbook, Part II. G Hunt CDL 55207 (C)
- 5. ICI Report on the 50 ton/week Runcol Plant, Flowsheet and Plant Item Summary, Report Number V/SPS/11 dated 9th November 1940.
- 6. An investigation of the factors governing the production of dark coloured Runcol. ICI Report No. R/GC/1288, 27th February 1943.
- 7. Sutton Oak Report SO/R/563. Impurities in production HT: estimate of the amount of Q, examination of distillation residues from plant HT. 29th December 1941.
- 8. Experimental Station Suffield, Alberta. Technical Minute No. 86. Stability of HT Thickened with Gelva and Plexiglas in BE/Chem 25-pdr. In Climatic Chamber. 25th February 1945.
- 9. Ptn/1252. (R.4078) Viscosity life of H.T.V.
- 10. Ptn/1252. (R.8421) Vinyl Acetate for Thickening H.T.
- 11. <u>www.nationalarchives.gov.uk</u> "Randle: history of charging department 1945". TNA:SUPP 5/1009
- 12. S.D. 129/B.34, Introduced by Amendment Leaflet (A.L.) No. 91 February 1942. Relevant amendments up to A.L. 148 incorporated in the reprint dated February, 1945. Drum, Storage, No. 6, Mk. I.
- 13. Drum Storage No. 6, Mark I. Drawing CD4451/1, dated 26 January 1945. Dstl/ESD/Ptn/657/706.
- 14. Porton Report No. 2655, Serial No. 86, Dated 23rd November 1944. Bomb A/C., L.C., 65 lb. charged vesicants. Report on Storage Trials carried out to determine the life of the weapon under Service conditions of storage. B.J. Perry and G.T. Lewis.
- 15. www.engnetglobal.com Definition of terne-plate.
- 16. Military Training Pamphlet No. 32 1940, The tactical and technical employment of chemical weapons.

- 17. Chemical Mine, Mk –BV., Drawing. C.D.4020/1B, dated 5.July 1935. Dstl/ESD/Ptn/657/911.
- 18. Chemical Weapons Movement History Compilation, WR Brankowitz, 27 April 1987. Office of the Program Mnager for Chemical Munitions (Demilitarisation and Binary) (Provisional), APG, Maryland, 21010-5401.
- 19. <u>www.nationalarchives.gov.uk</u> "Chemicals". War Office and Ministry of Supply; Contract Record Books. SUPP 4/342
- (a) Suffield Report No. 5. Coefficients of Expansion, Viscosities and Melting Points of HT and HTV of Various Alloprene Concentrations. 30th January 1942.
 (b) W.13276, Chief Superintendent, Porton. Letter dated 30th October 1942.
- 21. Chemical Warefare laboratories, Ottawa, Report No. 17. Purification of Levinstein HS by Solvent Extraction. 10th October 1944.
- 22. Memorandum to: Honourable C D Howe, PC, MP Minister of Munitions and Supply, Ottawa, March 7, 1943. Re: Production of HT Mustard Gas in Canada.
- 23. Department of National Defence Army, Chemical Warfare Laboratories. 28th March 1944, Re: HT Production, Stormont Chemicals.
- 24. "Behind the Mask." Todd Libou, Go! Magazine,

6 Acknowledgments

Sincere thanks are due to Chris Warde and Julie Livingston who carried out the task of searching through old documents in a systematic and conscientious manner. Eddy Doust also played an important role in sifting and sorting the documents found and directing the search effort. They are all to be congratulated on a job well done.

INTENTIONALLY LEFT BLANK

Initial distribution

International Research Office US Army Chemical Materials Agency Dstl Knowledge Services Author

File

1 Electronic Copy

3 Hard Copies, 1 Electronic Copy

2 Hard Copies, 1 Electronic Copy

1 Hard Copy

1 Hard Copy, 1 Electronic Copy

INTENTIONALLY LEFT BLANK

Report Documentation Form

A copy of this form is to be completed by the principal author for all Dstl reports. When complete, it is to be bound as the last numbered pages of the finished report.

1. Originators Report Nu	mber incl. Version No	Dstl/CR24879 V1	
2. Report Protective Mari UNCLASSIFIED	kings and any other mark	cings e.g. Caveats, Descrip	tors, Privacy markings
		Methods of Manufacture as Held at Pine Bluff Arsenal	
4. Title Protective Markin	ngs incl. any Caveats	UNCLASSIFIED	
5. Authors Dr. Richar	d Soilleux		
6. Originator's Name and Environmental Sciences Dstl Porton Down, Salisbury, Wilshire, SP4 0JQ		7. MOD Sponsor Name	e and Address
8. MOD Contract number	r and period covered	N/A	
9. Other Report Nos.	N/A		
10. Date of Issue 18 th June 2007	11. Pagina 24 pages p	tion	12. No. of References

13. Abstract (A brief (approximately 150 words) factual summary of the report)

This review has been undertaken for the US to establish the provenance of HT currently awaiting disposal at Pine Bluff Arsenal. Detailed descriptions of the HT process have been found together with some information concerning impurities. Much has been discovered on the construction of the Storage Drum, No. 1, Mark 6 and the Mine Chemical No 1 Mk I. It has not been possible to confirm the known shipment of HT in 1946 but a separate previously unknown shipment direct to Pine Bluff in 1943 has been identified. HT of British origin was used in Canadian field trials but no documents proving Canadian production of HT have been found in the UK. The US Army Chemical Materials Agency has recently discovered Canadian documents that show that the Cornwall facility did produce HT. It is likely that the HT shipped to the US from Cornwall was of Canadian origin made to UK specifications. However, HT manufactured in the UK may also have been stored at the Canadian plant so a British origin, at least in part, for the "Cornwall" HT cannot be ruled out.

14. Abstract Protective Marking including any Caveats

UNCLASSIFIED

15. Keywords/Descriptors (Authors may provide terms or short phrases which identify concisely the technical concepts, platforms, systems etc. covered in the report.

Mustard, HT, CW Production, CW demilitarisation, Pine Bluff Arsenal.

16. Report Announcem Abstracts)	nent (refers to title/abs	tract being included in accessions lists	s e.g. Defence Reports
Announcement of	this report is UNLIMI	TED	
If there are limitations can be announced (mo		of this report please indicate below th	e groups to whom it
Can be announced	to MOD and its Agend	cies	
Can be announced	to UK Defence Contra	actors	
Can be announced	to Overseas Defence I	Departments	
Other (please speci	fy)		
17. Report Availability	У		
UNLIMITED distr	ibution		
☐ No Release withou	t approval of the Relea	ase Authority	
If the above do not apprequest without further		low the groups to whom the report maks.	y be released upon
Can be released to	MOD and its Agencie	S	
	other UK Governmen		
Can be released to	UK Defence Contract	ors	
Can be released to	Overseas Defence De	partments	
Other (please spec	ify)		
18. Downgrading Insti	ructions (check as app	ropriate)	
This report may be	automatically downgr	raded to after years	
☐ This report may be		rs after publication	
19. Authorisation (Con			
19. Authorisation (Co.		2:	
	Name	Signature	Date
ESD Chief Scientist	Adrian Baker	Alfol	19/6/07
Project Manager	Eddy Doust	Cr 2	19/06/07.
Technical Reviewer	Dr. Robin Black	Russ	18/06/07

When complete the form is to be bound into the report to which it refers and is to form the last numbered pages of the report. Dstl Knowledge Services, Porton Down will enter an abstract and other details onto the relevant report management systems.

This page is intentionally blank